



Attorney Docket No. 75252-008

Serial No.: 09/776,656

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

Applicants: David Johnson et al.

Serial No.: 09/776,656

Filing Date: February 5, 2001

Title: Liquid, Radiation-Curable Composition, Especially for Stereolithography

Examiner: C. Hamilton

Group Art Unit: 1752

RECEIVED  
OCT 29 2003  
TC 1700

Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

**DECLARATION UNDER 37 C.F.R. § 1.131**

I, Frank Tran, state:

1. I am employed as a Research and Development Chemist by Vantico A&T US Inc., 5121 San Fernando Road West, Los Angeles, California 90039-1011.

2. I am familiar with Vantico A&T US Inc.'s patent application 09/776,656, claim 1 of which reads as follows:

- "1. A liquid, radiation-curable composition comprising:
- a) 40 to 80 percent by weight of one or more than one compound having at least two epoxy groups,
  - b) 0.1 to 10 percent by weight of a cationic photoinitiator or a mixture of cationic photoinitiators comprising a sulfonium salt,
  - c) 2 to 30 percent by weight of a (meth)acrylate compound having at least one hydroxy group,
  - d) 5 to 40 percent by weight of a hydroxy compound having no unsaturated groups,

e) 0 to 30 percent by weight of at least one liquid poly(meth)acrylate having a (meth)acrylate functionality of more than 2 and having no hydroxy groups,  
f) 0 to 40 percent by weight of at least one liquid cycloaliphatic or aromatic di(meth)acrylate having no hydroxy groups, and  
g) 0 to 10 percent by weight of a reactive diluent,  
wherein the sum of components a), b), c), d), e), f), and g) is 100 percent by weight, and components c), d), e), f) and g) are different, and the composition contains no free radical initiator selected from the group consisting of benzoines, acetophenones, benzil, benzil ketals, anthraquinones, triphenylphosphine, benzoylphosphine oxides, bisacylphosphine oxides, benzophenones, thioxanthenes, xanthenes, acridine derivatives, phenazine derivatives, quinoxaline derivatives, 1-phenyl-1,2-propanedione 2-O-benzoyl oxime, 1-aminophenyl ketones, 1-hydroxy phenyl ketones, and ionic dye-counterion compounds.”

3. On or about September 8 or 9 of 1999, I attended a meeting with my boss, Rich Leyden, and chemist David Johnson in Rich’s office. At that meeting, Rich assigned me the task of developing a liquid, radiation-curable composition with the same raw materials as our commercial product SL 5170 but to leave out the free radical photo initiator, and to make sure that this new composition would achieve equivalent or better mechanical property values compared to SL 5170.

4. In response to Rich’s instructions, I formulated a series of six compositions that I called S179-39A through S179-39F. S179 refers to the title of my lab notebook. Attached as Exhibit 1 is a complete and accurate copy of the pages from my lab notebook S179 that show the work that I performed in relation to formulations 39A through 39F.

5. On September 10, 1999, I began working on the project, as shown by the date at the top of my first notebook page (Exhibit 1, page 39). Also on September 10, 1999, I created a spreadsheet for recording the data from my project. As I ran the tests, I simultaneously recorded the data in the spreadsheet. The project spanned the period from September 10, 1999 to December 27, 1999. On March 25, 2003, I printed the completed spreadsheet. The printout is attached as Exhibit 2.

6. SL 5170 was the control group for these experiments. SL 5170 was a Ciba (now Vantico) commercial product in 1999, and it contained epoxy monomers, a cationic photoinitiator comprising a sulfonium salt, a hydroxy (meth)acrylate, a hydroxy compound having no unsaturated groups, and a free radical photoinitiator. Compositions 39A through 39F all contain the same epoxy monomers in the same amounts as the control SL 5170. In addition, compositions 39A through 39F all contain Tone 0301, a hydroxy compound having no unsaturated groups in the same amount as the control SL 5170. Compositions 39A through 39C contain the same cationic and free radical photoinitiators as SL 5170, but different (meth)acrylates. N3700 and SR 399 are (meth)acrylate compounds having at least one hydroxyl group. Composition 39D contains only the cationic photoinitiator (UVI 6974) and no free radical photoinitiator, but fails to contain a hydroxy (meth)acrylate. Compositions 39E and 39F contain two hydroxy (meth)acrylates and a cationic photoinitiator, but no free radical photoinitiator.

7. On page 41 of my lab notebook S179, I entered the data collected in window pane and flex modulus tests for the first composition of the series, 39A. In the window pane test, single layer, square specimens are created by photocuring the liquid composition with a HeCd laser. The thickness of the cured specimen is taken as a measure of the photosensitivity of the liquid resin. The plotting of the resulting layer thickness on a graph against the logarithm of the irradiation energy used gives a "working curve". The slope of the curve is termed  $D_p$ , which is defined as depth of penetration. The energy value at which the curve passes through the x-axis is termed  $E_c$ , which is defined as the critical energy. Together, the depth of penetration ( $D_p$ ) and the critical energy ( $E_c$ ) for initiation of polymerization, i.e., the energy at which gelling of the material takes place, define the photospeed.

8. I built window panes for each of the formulations 39A through 39F between September 10, 1999 and October 16, 1999. The following is a chart summary of my lab notebook pages showing the date on which the window pane for each composition 39A through 39F was built and tested.

Composition	Build and Test Date	Lab Notebook Page (Exhibit 1)
39A	September 10, 1999	41
39B	September 17, 1999	47
39C	October 14, 1999	69
39D	October 14, 1999	69
39E	October 16, 1999	72
39F	October 16, 1999	72

Exhibit 2 shows the results of the window pane tests for SL 5170 and 39A through 39F in the section labeled "Photospeed". In addition, Exhibit 3 is a collection of computer printouts showing the window pane data that I kept electronically for compositions 39A through 39F. The window pane data in Exhibits 2 and 3 was entered simultaneously with the window pane data entries in my lab notebook.

9. The green strength of compositions 39A through 39F was tested and determined by building so-called green models. A green model is a multi-layer shaped article built from a liquid, radiation-curable composition with the aid of radiation in which the article is not yet fully cured. The flexural modulus refers to the stiffness or resistance to bend of a material. The green strength is determined by measuring the flexural modulus (ASTM D790) of the model 10 and 60 minutes after imaging is complete. The flexural modulus is taken as an average of three separate measurements. The following is a chart summary of my lab notebook pages showing the date on which the green models were built and tested for each composition 39A through 39F.

Composition	Date tested	Lab Notebook Page (Exhibit 1)
39A	September 11, 1999	42
39B	September 18, 1999	47
39C	October 14, 1999	70
39E	October 15, 1999	70
39F	October 18, 1999	73
39F	October 18, 1999	73

Exhibit 2 shows the results of the green strength tests for SL 5170 and compositions 39A through 39F in the section labeled "Green Strength (MPa)". The green strength data in Exhibit 2 was entered simultaneously with the green strength data entries in my lab notebook.

10. My goal for the above compositions 39A through 39F was to test and compare the mechanical properties of the articles produced by the compositions, and in particular compositions 39E and 39F in which I removed the free radical photo initiator, so as to isolate and test the affect of the hydroxy acrylate in the solution.

11. When I ran the window pane tests on October 16, 1999, I realized that composition 39F had a photospeed equivalent to SL 5170 even though composition 39F did not contain a free radical photo initiator. When I tested the green strength of compositions 39E and 39F on October 15 and 18 of 1999 respectively, I realized that both compositions 39E and 39F produced parts having a green strength as good as or better than SL 5170 even though both compositions 39E and 39F failed to contain a free radical photo initiator. It was at this time that I appreciated and recognized that parts could be built from compositions 39E and 39F that exhibited equivalent or better mechanical property values as compared to SL 5170.

12. During the time I formulated and tested compositions 39A through 39F, I was also busy working on two other major projects in the lab. I had to prioritize and divide my time according to the demands of all three projects.

13. I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that the statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under §1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

6-6-03  
Date

  
Frank Tran